

Integran Technologies Inc.

Claims

1. Process for cathodically electrodepositing a selected metallic material on a permanent or temporary substrate in nanocrystalline form with an average grain size of less than 100 nm at a deposition rate of at least 0.05 mm/h, comprising:
providing an aqueous electrolyte containing ions of said metallic material, agitating the electrolyte at an agitation rate in the range of 0.0001 to 10 liter per min and per cm² anode or cathode area or at an agitation rate in the range of 1 to 750 milliliter per min and per Ampere, and passing single or multiple cathodic-current pulses between said anode and said cathode.
2. Process according to claim 1, wherein a duty cycle is in a range of 5 to 100%.
3. Process according to any of claims 1 to 2, wherein a frequency of the cathodic-current pulses is in a range of 0 to 1000 Hz.
4. Process according to any of claims 1 to 3, wherein the single or multiple cathodic-current pulses between said anode and said cathode have a peak current density in the range of about 0.01 to 20 A/cm².
5. Process according to claim 4, wherein the peak current density of the cathodic-current pulses is in the range of 0.1 to 20 A/cm² preferably in the range of 1 to 10 A/cm².

6. Process according to any of claims 1 to 5, wherein said selected metallic material is (a) a pure metal selected from the group consisting of Ag, Au, Cu, Co, Cr, Ni, Fe, Pb, Pd, Rt, Rh, Ru, Sn, V, W, Zn, or (b) an alloy containing at least one of the elements of group (a) and alloying elements selected from the group consisting of C, P, S and Si.
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7. Process according to any of claims 1 to 6, wherein a $t_{\text{cathodic-on}}$ -time period is in the range of 0.1 to 50 msec, a $t_{\text{cathodic-off}}$ -time period is in the range of 0 to 500 msec and a $t_{\text{anodic-on}}$ -time period is in the range of 0 to 50 msec.
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8. Process according to any of claims 2 to 7, wherein the duty cycle is in the range of 10 to 95 %.
9. Process according to any of claims 1 to 8, wherein the deposition rate is at least 0.075 mm/h and preferably at least 0.1 mm/h.
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10. Process according to any of claims 1 to 9, which comprises agitating the electrolyte at an agitation rate in the range of 1 to 500 milliliter per min and per Ampere.
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11. Process according to any of claims 1 to 10, which comprises agitating the electrolyte by means of pumps, stirrers or ultrasonic agitation.
12. Process according to any of claims 1 to 10, which comprises a relative motion between anode and cathode.
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13. Process according to claim 12, wherein the speed of the relative motion between anode and cathode ranges from 0 to 600 m/min, preferably in the range from 0.003 to 10 m/min.
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14. Process according to any of claims 12, wherein the relative motion is achieved by rotation of anode and cathode relative to each other.
15. Process according to claim 14, wherein a rotational speed of rotation of anode and cathode relative to each other ranges from 0.003 to 0.15 rpm and preferably between 0.003 to 0.05 rpm.
16. Process according to claim 12 to 13, wherein the relative motion is achieved by a mechanized motion generating a stroke of the anode and the cathode relative to each other.
17. Process according to claim 12 or 16, wherein the anode is wrapped in an absorbent separator.
18. Process according to any of claims 1 to 17, wherein said electrolyte contains a stress relieving agent or a grain refining agent selected from the group of saccharin, coumarin, sodium lauryl sulfate and thiourea.
19. Process according to any of claims 1 to 18, wherein said electrolyte contains particulate additives in suspension selected from pure metal powders, metal alloy powders or metal oxide powders of Al, Co, Cu, In, Ng, Ni, Si, Sn, V and Zn, nitrides of Al, B and Si, carbon C (graphite or diamond), carbides of B, Bi, Si, W, or organic materials such as PTFE and polymers spheres, whereby the electrodeposited metallic material contains at least 5 % of said particulate additives.
20. Process according to claim 19, wherein the electrodeposited metallic material contains at least 10 % of said particulate additives.
21. Process according to claim 19, wherein the electrodeposited metallic material contains at least 20 % of said particulate additives.

22. Process according to claim 19, wherein the electrodeposited metallic material contains at least 30 % of said particulate additives.
- 5 23. Process according to claim 19, wherein said electro deposited metallic material contains at least 40 % of said particulate additives.
24. Process according to any of claims 19 to 23, wherein the particulate additives average particle size is below 10 μm preferably below 1000 nm, preferably below 500 nm, preferably below 100 nm and most preferably below 100 nm
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